

(12) **UK Patent Application** (19) **GB** (11) **2 194 791** (13) **A**

(43) Application published 16 Mar 1988

(21) Application No **8715623**

(22) Date of filing **2 Jul 1987**

(30) Priority data

(31) **5357/86**

(32) **14 Jul 1986**

(33) **CS**

(71) Applicant

Vysoka Skola Chemicko-Technologiccka

(Incorporated in Czechoslovakia)

Praha, Czechoslovakia

(72) Inventors

Vladimir Kubanek

Jaroslav Kralicek

Budimir Veruovic

Zdenek Cimburek

(74) Agent and/or Address for Service

Saunders & Dolleymore,

2 Norfolk Rd, Rickmansworth, Herts WD3 1JH

(51) INT CL⁴

C08J 3/20 // A61K 7/46

(52) Domestic classification (Edition J):

C3L 408 412 414 DA DN

C3W 111 205 215 217 218

U1S 1340 3015 3016 C3L

(56) Documents cited

GB A 2173208

GB 1126108

US 3553296

GB 1538085

US 3661838

(58) Field of search

C3L

C3V

Selected US specifications from IPC sub-classes C08J

C08L

(54) **Incorporating scenting material into plastics**

(57) For scenting of products made of plastics the scenting material is first sorbed on to a powdery, porous carrier material in an amount of 1 to 70% by weight, and may be pressed to granules in the presence of a binding agent, the obtained immobilised powdery or granulated scenting material is mixed in an amount of 1 to 30% by weight, related to the weight of the plastics, with the plastics, the homogenous mixture is thereafter processes by pressing, injection moulding, extruding or blow moulding.

In a typical Example (2), polybutylmethacrylate is dissolved in a scenting composition and the mixture absorbed on silica; this product is then blended with polyethylene.

BEST AVAILABLE COPY

GB 2 194 791 A

SPECIFICATION

Method for the scenting of products made of plastics

- 5 The invention relates to a method for the scenting of products made of plastics, which products should retain their scent for a long time.
- 10 Products from plastics are at present used in many branches of human activity. Due to their specific properties, such as toughness, strength, chemical resistivity, hygienic unobjectionability, esthetic appearance and required
- 15 colour, products from plastics have acquired an irreplaceable place in industry and home. They are mostly used as parts of kitchen furniture, for the manufacture of kitchen utensils and hygienic and durable packagings, or in
- 20 industry as parts of products, for instance of electric appliances, parts of motor cars and the like.
- The extensive use of products made from plastics, particularly in homes, brought with it
- 25 also the possibility of their scenting. Due to the scenting of toys for children, of decorative products from plastics and of wrapping materials, the extent of their application has increased. The application of scenting processes
- 30 to products made from plastics resulted in the manufacture of new and original products from plastics.
- The scenting of products made from plastics has been performed substantially by two
- 35 methods. The first method consists in that the plastics, which is generally in the shape of granules, is mixed with the required amount of scenting material (1 to 10 % by weight) and is subsequently processed. This method is
- 40 rather uneconomical as the major part of the scenting material is evaporated already in the course of processing or is heat degraded; in most cases the scenting material leaves the processing machine without being combined
- 45 with the polymer. The scenting material is deposited on the surface of the products because it is incompletely built into the polymer matrix. The product is on the surface "greasy", unsightly and deformed.
- 50 In the second method the polymer is mixed with the scenting material at a ratio of 1:1.5 to 1:2 by weight and the mixture is processed in an extruding machine into granules containing up to 50 % by weight of the scenting
- 55 material. This product is usually called Master Batch. The required amount of the Master Batch is thereafter added to the polymer prior to its processing to the final scented product. A drawback of this method is again the large
- 60 loss of the expensive scenting material in the manufacture of the Master Batch. These losses are reflected in the price of the Master Batch which is substantially higher than the price of the scenting material, although Master
- 65 Batch contains usually about 50 % of the

70 scenting material. Another drawback of this method is that the scenting material is exposed to high temperatures twice. First in the course of manufacture of the Master Batch, and then in the course of its working-in into the product. Due to the substantial stress of the scenting material, its degradation and changes of its original characteristic scent take place.

- 75 It is an object of this invention to eliminate, or at least to substantially mitigate, said drawbacks of existing methods of scenting of products made from plastics, particularly of plastics based on polyethylene, polypropylene,
- 80 polystyrene, cellulose derivatives, acrylonitril-butadiene-styrene copolymers, by a method according to the invention, which consists in that the scenting material is sorbed on to a pulverious porous carrier material in an amount
- 85 of 1 to 70 % by weight, alternatively is pressed into granules in the presence of a binding agent, the thus obtained immobilised powdery or granulated scenting material is
- 90 mixed in an amount of 1 to 30 % by weight, related to the weight of the plastics, with the plastics, the homogenous mixture is processed by pressing, injection moulding, extruding or blow moulding. The scenting material is
- 95 sorbed on to the powdery porous carrier material, the specific surface of which is 0.1 to 800 m²/g and is prepared from polymers and copolymers based on polyethyleneterephthalate, polyphenylene oxide, polypropylene, polyethylene, polyamides, derivatives of cellulose,
- 100 acrylonitrilbutadiene-styrene, hydrated oxide of silicon, titanium, aluminium, bentonite, possibly celite and their mixtures. The powdery porous carrier material with the scenting material is pressed into granules in the presence of 1 to
- 105 15 % by weight of a binding agent, related to the weight of the scenting material, the binding agent used being based on polybutylmethacrylate, polypropylmethacrylate, polyvinylacetate, polystyrene, cellulose derivatives,
- 110 possibly butylmethacrylate- and methylmethacrylate-, vinylacetate- and styrene-, vinylacetate-copolymers.

The immobilisation of the scenting material according to this invention is performed by mixing the scenting composition with the powdery porous carrier material. The sorption of the scenting material on the carrier material

115 proceeds at normal temperature. The size of particles is from 20 to 50 µm. The carrier materials must not be soluble in the respective scenting composition. A main requirement is that the carrier material should be capable of good connection with the chemical structure of the plastics. Due to the large specific surface of the mentioned carrier types of scenting materials and their porous structure, they act as active filler agents in plastics and can easily be coloured with colours similar to those used for other plastics.

- 120
- 125
- 130 The processing of plastics with immobilised

scenting material is accomplished by current technological processes and on currently used equipment designed for the pressing of plastics, for injection moulding, extruding and blow moulding. The conditions of processing are also the same as for plastics without scenting material. In the course of processing of plastics with immobilised scenting material a dispersion of the scenting material in the polymeric matrix is accomplished, from which it is released very slowly. The speed of release of the scenting material from the polymeric matrix is determined by the speed of its diffusion in the polymeric matrix. The content of the scenting material in the final product varies, according to requirements on the intensity of the scent, from 1 to 10 % by weight. The time interval within which the product retains the scent depends not only on the type of the scenting material and on its concentration, but also on the kind of plastics and on the wall thickness of the product. For instance a polyethylene wrapping foil containing 5 % by weight of a scenting material releases the scent for 14 weeks, whereas a 3 mm thick polyethylene plate with the same content of scenting material smelled pleasantly over the whole period of testing, in the case given over 13 months. The presence of the scenting material with up to 5 % by weight in the product of plastics influences neither its physical properties, nor its colour shade, nor the colouring process. The immobilised scenting material can be stored for an unlimited period, particularly when stored in a closed vessel. The scenting material prepared according to this invention can be considered to be a scenting material in solid state and the technology of its processing can be compared with the technology used for the colouring of plastics.

The method according to this invention enables scent or any kind of aroma, such as forest scent, fruit scent, deodorant or other scents, to be incorporated in plastics products for domestic use such as cups, trays, soap and tooth brush holders, Christmas trees, artificial flowers, flower pots, combs, children's toys, ornamental products, wrapping material for fruit, linen and the like. The scenting material becomes part of the product from which it is gradually released and endows the product with a long-lasting pleasant odour. The long-lasting pleasant smell of products made of plastics according to this invention is afforded by formation of equilibrium-association products of the scenting composition with the polymeric structure of the plastics. The scenting material sorbed on the porous carrier material is by this process protected from the heat effect, i.e. it is heat-stabilised. This circumstance allows the application of scenting compositions for the scenting of plastics which are sensitive to heat at which the plastics are processed. Due to that the heat

degradation of the scenting material is minimised. This immobilisation of the scenting materials for the scenting of plastics limits also their losses and the original scent of the scenting material is maintained. No bleeding of the scenting material on the surface of products has been observed. The scenting of products made from plastics according to this invention is from the point of view of economy substantially more advantageous than presently used methods.

A method for the scenting of plastics products will now be described in greater detail by way of examples.

Example 1

20 g of polyethyleneterephthalate in powdery porous shape having a specific surface of 80 m²/g were mixed with 20 g of a scenting composition based on eucalyptus oil, benzoate and toluate derivatives, terpinylacetate, dehydrolinalacetate and aldehydes and ketones and their derivatives. The obtained powdery material, which represents the immobilised scenting material, was added to 940 g of polypropylene granules and thoroughly mixed in a mixer. In the course of this process the polypropylene granules were covered by the powdery material i.e. by the stabilised scenting material and their mutual separation did not take place for a long time. The thus prepared mixture was processed on an extrusion machine at a temperature of 200 °C and rods of a diameter of 4 mm and of a length of 200 mm were formed. The rods had a smooth, shiny and dry surface with the characteristic smell of the scenting material. The intensity of the smell did not change for the whole test period of 20 months.

Example 2

7 g of polybutylmethacrylate with a melting index of 23 were dissolved in 50 g of a scenting composition based on limonen, dipenten and veratren and the thus prepared solution was mixed with 50 g of powdery porous silicon oxide with a specific surface of 120 m²/g. A homogenous powdery material was obtained by this process, from which cylinders of a diameter of 3 mm and of a length of 5 mm have been pressed. The cylinders had a sufficient mechanical strength so that they remained intact during normal manipulation. 100 g of said cylinders were mixed with 900 g of granulated polyethylene in a mixer and the mixture was processed on an extruder to tubes of an internal diameter of 4 mm and of an external diameter of 6 mm. The tubes retained their original characteristic scent of the scenting composition for the test period of 13 months.

Example 3

30 g of a scenting composition of the citrus type were mixed with 30 g of powdery po-

- rous polyphenylene oxide with a specific surface of 560 m²/g. The thus prepared powdery material represents an immobilised scenting material on a carrier material which material
- 5 was further mixed in a mixer for 30 minutes with 940 g acrylonitril-butadiene-styrene-copolymer. The prepared mixture showed a homogenous distribution of the powder material on the surface of granules. This mixture
- 10 remained stable for the test period of 30 minutes. Strings of a diameter of 2 mm were pressed on an extruder from the thus prepared mixture at a temperature of 230°C. After storage of said strings for 14 months
- 15 no loss of intensity of the original scenting composition was observed.

Example 4

- 40 g of powdery porous acrylonitril-butadiene-styrene-copolymer with a specific surface of 34 m²/g was mixed with 40 g of a scenting composition of the citrus type. A granular material was prepared therefrom which was mixed with 960 g of polyethylene granules, and a foil was made therefrom by blowing. The thus prepared foil maintained its original characteristic scent of the used scenting composition for 16 weeks.

Example 5

- 8 g of butylmethacrylate-(84 % by weight)-methylmethacrylate-copolymer was dissolved in 60 g of a scenting composition based on terpene hydrocarbons and alcohols. The solution of the copolymer in the scenting composition was gradually mixed with 60 g of hydrated powdery silicon oxide with a specific surface of 120 m²/g. Granules of a diameter of 2 mm and of a length of 5 mm were prepared from the thus obtained powdery mixture on a manual press. The prepared granules were added to 880 g of tough polystyrene of a melting index of 4 and after thorough mixing of the mixture test plates were pressed which
- 45 has the same smell of the used composition. The intensity of this refreshing scent was constant for a test period of 18 months.

CLAIMS

- 50 1. Method for the scenting of products made of plastics in which the scenting material is sorbed on to a powdery, porous carrier material at an amount of 1 to 70 % by weight, and may be pressed to granules in the
- 55 presence of a binding agent, the obtained immobilised scenting material in an amount of 1 to 30 % by weight, related to the weight of the plastics, is mixed with the plastics, and the homogenous mixture is processed by
- 60 pressing, injection moulding extruding or blow moulding.
2. Method according to Claim 1 in which the scenting material is sorbed on to a powdery, porous carrier material, the specific surface of which is from 0.1 to 800 m²/g and

- which is made of polymers and copolymers based on polyethyleneterephthalate, polyphenyleneoxide, polypropylene polyethylene, polyamides, cellulose derivatives, acrylonitril-butadiene-styrene, hydrated silicon dioxide, titanium dioxide, aluminium oxide, bentonite, celite or their mixtures.
- 70 3. Method according to Claim 1, in which the powdery porous carrier material with the scenting material is pressed to granules in the presence of 1 to 15 % by weight of a binding agent, related to the weight of the scenting material, the binding agent being based on polybutylmethacrylate, polyisopropylmethacrylate, polyvinylacetate, polystyrene, cellulose derivatives, possibly butylmethacrylate- and methylacrylate-, vinylacetate- and styrene-, vinylacetate- and butylmethacrylate-copolymers.
- 80 4. A method for the scenting of products made of plastics substantially as described in any one of the Examples 1 to 5.

Published 1988 at The Patent Office, State House, 66/71 High Holborn, London WC1R 4TP. Further copies may be obtained from The Patent Office, Sales Branch, St Mary Cray, Orpington, Kent BR5 3RD. Printed by Burgess & Son (Abingdon) Ltd. Con. 1/87.

BEST AVAILABLE COPY